



# Research on the aging and rejuvenation mechanisms of asphalt using atomic force microscopy

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## HIGHLIGHTS

- The aging and rejuvenation mechanisms of asphalt binders were investigated from macroscopic and microscopic scales.
- The type and content of rejuvenator does not militate on the microstructures of rejuvenated asphalt binders.
- The rejuvenator must be evaluated from the perspective of actual performances of rejuvenated asphalt binders.

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## ABSTRACT

To investigate the mechanism of aging and rejuvenation of asphalt, traditional laboratory and atomic force microscopy (AFM) tests were conducted for asphalt specimens, respectively. Experimental results indicated that the penetration and ductility of virgin asphalt significantly decreased after aging. These results could not satisfy the technical requirements of the specification. The softening point and viscosity of virgin asphalt increased due to aging process. Rejuvenator had a significant impact on the performance of aged asphalt binders. The penetration of aged asphalt binders conspicuously increased with an increase in the rejuvenator content. The softening point and viscosity of aged asphalt binders obviously decreased with the rejuvenator content increasing, and the optimum rejuvenator content was about 12% by mass in this study. Through comparing virgin and aged asphalt binders via AFM test, it confirmed that light components contents of asphalt binders greatly decreased during the aging process. The aging effect of asphalt binder was a sluggish process, and the content and microstructure of its internal components would vary with the altering of aging degree. The rejuvenator did not recompose the composition of aged asphalt binders. The results showed that the rejuvenation process of rejuvenator was a physical interaction, and the kinds and contents of rejuvenator would not alter the microstructure of recycled asphalt binders.

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## 1. Introduction

In recent years, reclaimed asphalt pavements (RAP) are increasing annually in China. Large amount of RAP not only occupy a lot of land, but also give rise to environmental issues [1,2]. On the other hand, limited virgin aggregates and the rise in the price of raw materials have seriously constrained the rehabilitation and construction of pavement industry [3]. RAP has been attracting unprecedented attention on account of its economic and environmental performance [4].

The aging of asphalt binders is an important cause for road performance degradation. The performance restoration of aged asphalt binders is an important goal for asphalt pavement recycling

technology [5]. Therefore, studying the mechanisms of aging and rejuvenation of asphalt has crucial significance to advance the asphalt pavement recycling technology.

To investigate the mechanisms of aging and rejuvenation of asphalt, numerous research studies have been conducted for asphalt from various perspectives [6–9]. In consideration of many studies focused on the once asphalt aging and rejuvenation, Zhang et al. [10] carried out the repeated aging and rejuvenation tests for asphalt in the RAP. Upon the test data, the performance change of asphalt presented three stages during repeated asphalt aging and rejuvenation tests, and the results demonstrated the non-unilateral changes of the performance of the repeated asphalt aging and rejuvenation. Cucalon et al. [11] analyzed the effects and mechanisms of rejuvenation through extensive rheological and physicochemical characterization by considering experimental variables such as type of recycling agent, asphalt type, and so on.

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The results showed that rejuvenation was effective to reduce the midpoint and end of glass transition temperature. Chemical characterization was not conclusive, and yet the results were interpreted as strong recycling agent-asphaltene polar interaction. The rheological benefits from rejuvenation were gradually lost during long-term aging process. Bai [12] investigated the low temperature properties of the aged styrene-butadiene styrene (SBS) modified asphalt binder with/without rejuvenators using traditional methods. The results indicated that the aging declined low temperature properties of SBS modified asphalt binder, and the rejuvenators improved the low-temperature properties of aged SBS modified asphalt binders. The degree of improvement increased with an increase in rejuvenator content. Cooper et al. [13] characterized the laboratory performance of traditional asphalt mixtures and mixtures containing RAP and/or recycled asphalt shingles (RAS), with and without recycling agents (RAs). Results indicated that the use of RAs improved the blending between aged and virgin asphalt binders, which adversely affected the intermediate- and low-temperature performances of asphalt mixtures.

As research continues, the relevant studies have entered microscopic level. In the microscopic research field, some researchers observed the interface morphology and structural feature of asphalt materials by Atomic Force Microscopy (AFM) [14–17]. De et al. [18] analyzed the microstructure detail of asphalt by thermal phase detection AFM. Phase contrast and topography images showed that the asphalt morphology was highly dependent on temperature. The 'bee-like structure' changed considerably at temperatures between 50 °C and 56 °C. Guo et al. [19] adopted the Peak Force QNM mode of AFM to characterize the morphology and mechanical property of asphalt binder at different distances to filler surface. The results demonstrated that AFM could characterize the interfacial interaction between asphalt binder and mineral aggregate. Rossi et al. [20] evaluated the effect of additives on the asphalt structure and compared with pristine binder as a reference by microstructural investigations carried out via AFM and fundamental rheological tests. In addition to the research studies mentioned above, AFM is also utilized to explore the effects of different warm mix asphalt (WMA) additives on the nanostructure and microstructure as well as the adhesive and cohesive properties of asphalt binders [21].

The aforementioned research studies exhibit that the investigation of aging and rejuvenation mechanisms of asphalt has become a research hotspot and that AFM is an effective technology to observe the micromorphology of asphalts. However, the asphalt aging and rejuvenation has been mainly researched from the macroscale level, but less focused on the microscopic level, especially analyzing the microstructure morphology of asphalt by AFM. Thus, in this paper, the asphalt aging and rejuvenation mechanisms are further investigated based on traditional laboratory and AFM tests, respectively.

## 2. Objective and scope

The objectives of this study are to investigate the aging and rejuvenation mechanisms of asphalt binders from macroscopic and microscopic scales, respectively. In order to achieve this goal, the three main elements are the followings:

- (1) To implement traditional laboratory tests, e.g. penetration test, softening point test, ductility test and viscosity test, for analyzing the effects of aging on the macroscopic performances of asphalt binders.
- (2) To explore the effects of different rejuvenator contents on performances of aged asphalt binders, and determine the optimal rejuvenator content in this study.

- (3) To reveal the influence of aging and rejuvenation effects on the microstructure of asphalt binders via AFM.

## 3. Experiments

### 3.1. Raw materials and technical properties

Virgin asphalt binder with the penetration grade of 70# was selected as base material in this research. Seventy means the penetration of the asphalt binder at 25 °C is ranging from 60 to 80 (units in 0.1 mm) according to the Chinese specification, JTG F40-2004 [22]. The properties of asphalt binders were measured according to the test methods of specification Standard Test Methods of Bitumen and Bituminous Mixtures for Highway Engineering, JTG E20-2011 [23], as shown in Table 1. RA100 and RA102 were adopted as the rejuvenators, which was produced by Sobute New Materials Co., Ltd. The physical properties of rejuvenators are presented in Table 2.

### 3.2. Specimen fabrication and test methods

#### 3.2.1. Specimen fabrication

In general, the long-term aging asphalt is simulated by pressure aging vessel (PAV). Unfortunately, the test apparatus of PAV is broken in our laboratory. According to the reference [24], if the aged time is a good choice, the thin Film Oven Test (TFOT) can be used to simulate long-term aging. Therefore, the TFOT tests were carried out under 163 °C ± 0.5 °C for 15 h to fabricate aged asphalt specimens for traditional laboratory and AFM tests.

#### 3.2.2. Traditional laboratory tests

Traditional laboratory tests, penetration test, softening point test, ductility test and viscosity test, were performed for virgin asphalt binders, aged asphalt binders and rejuvenated asphalt binders with 6%, 8%, 10% and 12% rejuvenator contents by mass. The tests are designed to inspect the altering of macroscopic performance of asphalt binders caused by the aging and rejuvenation effects. The details of test methods are shown in specification Standard Test Methods of Bitumen and Bituminous Mixtures for Highway Engineering, JTG E20-2011 [22].

#### 3.2.3. Asphalt microstructure observation tests by AFM

There have been a great number of research studies in asphalt binders and mastics using various microscopic analysis

**Table 1**  
Properties of the virgin asphalt binders used.

Property indices	Test results	Test methods
Penetration (25 °C, 5 s, 100 g)/0.1 mm	63	T0604–2011
Softening point/°C	48	T0606–2011
Ductility, 10 °C/cm	29	T0605–2011
Viscosity, 135 °C/mPa s	498	T0625–2011

**Table 2**  
Physical properties of the rejuvenators used.

Property indices	Test results		Test methods
	RA100	RA102	
Flash point/°C	224	255	T0611–2011
Viscosity ratios before and after RTFOT	1.62	1.2	T0610–2011, T0621–2011
Mass ratios before and after TFOT	1.03	0.1	T0610–2011
Saturates (%)	22.5	19.2	T0618–1993
Aromatics (%)	65.8	71.0	T0618–1993
Density, 15 °C/(g/cm <sup>3</sup> )	1.01	0.99	T0706–2011

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